PATENT

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for

RESONANT WAVEGUIDE-GRATING FILTERS AND SENSORS AND METHODS FOR MAKING AND USING SAME

by

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BACKGROUND OF THE INVENTION

This application claims priority to provisional patent application Serial No. 60/163,705 filed November 5, 1999, the entire text of which is specifically incorporated by reference herein without disclaimer. This application also claims priority to provisional patent application Serial No. 60/164,089 filed November 6, 1999, the entire text of which is specifically incorporated by reference herein without disclaimer.

1. Field of the Invention

The present invention relates generally to the field of optical filters and sensors. More particularly, it concerns the use of the guided-mode resonance effect occurring through the use of waveguide gratings attached to the endfaces of waveguides such as optical fibers in fields such as optical sensing and communications.

2. Description of Related Art

Resonance anomalies occurring in waveguide gratings (WGGs) have been the subject of current interest for spectral filtering applications [Magnusson and Wang, 1992; Wang and Magnusson, 1993; Wang and Magnusson, 1994; Shin *et al.*, 1998; Tibuleac and Magnusson, 1997; Tibuleac, *et al.*, 2000; Wawro, *et al.*, 2000; Avrutsky, *et al.*, 1989; Boye and Kostuk, 1999; and Rosenblatt, *et al.*, 1997]. Guided-mode resonances (GMRs) occurring in subwavelength WGGs admitting only zero-order propagating diffraction orders yield spectral filters with unique properties such as peak reflectances approaching 100%, narrow linewidths, and low sidebands. Filter characteristics, such as center wavelength, linewidth and sideband behavior, are defined by the waveguide-grating parameters, such as grating period, grating profile, refractive indices, layer thicknesses, and grating fill factor.

Changes in any parameters of the diffractive structure can result in a responsive shift of the reflected or transmitted wavelength band. In general, for spectral filtering applications, the most stable GMR structure is sought to prevent an unwanted resonance shift due to small parameter fluctuations. However, for spectroscopic sensing applications, it is desirable to enhance the resonance instability to create a device that will

respond to very small parameter changes. This type of device can be utilized, for example, to detect very small changes in the refractive index or thickness of a media being evaluated in biomedical, industrial or environmental sensing applications. Implementation of the guided-mode resonance effect for optical sensing using planar waveguide grating structures and free-space propagating incident waves has been proposed in previous publications [Wang and Magnusson, 1993; Shin *et al.*, 1998].

Experimental fabrication of waveguide gratings utilizing the GMR effect has primarily been restricted to planar WGGs with an incident beam that is propagating in free space. Experimental results for 1-D grating GMR filters incorporate single layer and multilayer reflection filter designs, including a TM polarization reflection filter utilizing the Brewster effect [Magnusson, *et al.*, 1998]. Double layer GMR filter efficiencies as high as 98.5% have been reported by Liu, *et al.* for TE incident polarization [Liu, *et al.*, 1998]. GMR crossed grating structures (2-D grating filters) have been experimentally fabricated by Peng and Morris [Peng and Morris, 1996], with a reported filter efficiency of 60%. Norton *et al.* [Norton, *et al.*, 1998] investigated the dependence of lineshape and tunability in central wavelength and resonant angle position on grating parameters.

Chen [Chen, 1988] reports a theoretical design incorporating a diffraction grating on an optical fiber endface that is used to excite higher order modes in multimode optical fibers. Wang *et al.* [Wang, *et al.*, 1995] reports a fiber optic proximity sensor design incorporating a diffraction grating on a fiber endface. However, the diffraction gratings reported in these two references do not have waveguide properties, and, consequently, do not exhibit the GMR effect.

A biosensor is an analytical device that integrates an immobilized biologically sensitive material (analyte), such as enzyme, antibody, DNA, cells, or organic molecules, with an electrochemical, piezoelectric, optical or acoustic transducer to convert a biochemical response into a signal for measurement, interpretation, or control. Electrochemical and optical sensors are most widely used. Optical biosensors can provide fast, accurate, and safe analyte detection. Current fiber-optic sensor technology

applies fluorescence, total internal reflection, intensity reflection, and surface-plasmon resonances.

The surface plasmon resonance (SPR) effect, is a widely used optical detection method that is highly sensitive to changes in the optical properties (refractive index, monolayer thickness) at the sensor surface. The term surface plasmon (SP) refers to an electromagnetic field charge-density oscillation that can occur at the surface of a conductor. An SP mode can be resonantly excited by parallel-polarized (TM) incident light. Conventional surface plasmon sensors include a prism or diffraction grating for phase matching of the incident and SP waves; commercial systems employ bulk optical components. Fiber-optic SPR sensors have been reported; in these a metal sleeve is deposited on the side of the fiber to which the analyte is contacted. A drawback of the SPR technology is the inherently large linewidth; typically $\Delta\lambda \sim 50$ nm. Therefore, a sensor utilizing the GMR effect that would provide smaller linewidths would exhibit a significant resolution dynamic-range advantage over SPR sensors.

SUMMARY OF THE INVENTION

In one respect, the invention is a waveguide grating device. The device includes at least one waveguide that has an end, and the end has an endface. As used herein, "waveguide" means any device possessing a structure capable of confining optical energy. As used herein, "endface" means a face on the end of a waveguide that may be oriented at any angle with respect to a wave being propagated through the waveguide. The device also includes a waveguide grating fabricated on the endface of the at least one waveguide. The waveguide grating has at least one waveguide layer and at least one grating layer. As used herein, "grating layer" includes any suitable layer possessing a grating. The gratings on the present grating layers include surface-relief type gratings (e.g., those in which the amplitude of the grating may be modulated) and volume gratings (e.g., those in which the refractive index of the grating may be modulated). The periodicity of the gratings of the present grating layers may be varied and/or their modulation depth (amplitude or index) may be varied. The grating may be nonuniform. As used herein, "waveguide layer" includes any suitable layer possessing a structure

capable of confining optical energy. Throughout the present disclosure, including the claims, waveguide layers are distinct from the waveguides on which they are fabricated. The at least one waveguide layer and the at least one grating layer may be the same layer.

In other respects, the at least one waveguide may be a fiber. The at least one waveguide may possess any suitable shape, including elliptical. The shape may be rectangular. The at least one waveguide may be a channel waveguide. The at least one waveguide may be cylindrical in shape. The at least one waveguide may be a slab waveguide. The at least one waveguide may be a ridge waveguide. The at least one grating layer may include a dielectric material. The at least one grating layer may include a glass. The at least one grating layer may include a polymer. The at least one grating layer may include a solid or liquid crystalline material. The at least one grating layer may include a semiconductor material. The at least one grating layer may include a photorefractive material. The at least one waveguide layer may include a dielectric material. The at least one waveguide layer may include a glass. The at least one waveguide layer may include a polymer. The at least one waveguide layer may include a solid or liquid crystalline material. The at least one waveguide layer may include a semiconductor material. The at least one waveguide layer may include a photorefractive material. The at least one grating layer and the at least one waveguide layer may be the same layer. The at least one grating layer and the at least one waveguide layer may be different layers in contact with each other. The waveguide grating may also include at least a third layer in contact with the at least one waveguide layer, the at least one grating layer, or both the at least one waveguide layer and the at least one grating layer. The at least third layer may be a buffer layer, which may be formed from any material suitable for forming either the at least one waveguide layer or the at least one grating layer, and which may be formed using the same techniques that may be used to form either the at least one waveguide layer or the at least one grating layer.

As a buffer layer, the at least third layer may be made of a dielectric and may serve to shape the spectral reflection of the waveguide grating, such as to lower the sidebands, shift the resonance to a desired wavelength, or narrow or widen the linewidth of the GMR. The buffer layer may serve as neither a waveguide layer nor a grating layer.

The at least third layer may also be formed of metal, which in some embodiments, may serve a buffer layer intermediate two layers of the waveguide grating that do not otherwise attach well to one another. In other embodiments, the at least third layer (made from either a dielectric or a metal, for example), may be the layer of the waveguide grating in contact with a substance to be sensed/evaluated. In some cases, the substance to be sensed may not attach itself efficiently to dielectric materials composing, at least in part, the waveguide grating. The use of a third layer that is thin may facilitate the attachment of substances being sensed. In still other embodiments, such as biomedical applications, an organic substance being sensed may attach itself only to other organic substances, and not to dielectric or metallic layers of the waveguide grating. In such situations, the use of a third layer (metallic, for example) may be used to which another (fourth) organic layer could be attached. The organic substance being sensed could then attach itself to such a fourth organic layer. The at least third layer may be distinct from both the at least one waveguide and grating layers. The waveguide grating may also include at least a third layer in contact with the at least one grating layer, and may include an arbitrarily large number of layers, each of which may be either additional waveguide layers, additional grating layers, or additional buffer layers.

In another respect, the invention is a system for spectral filtering and the system utilizes a guided-mode resonance effect in a waveguide. The guided-mode resonance effect is described below in greater detail. The system includes a waveguide grating device. The waveguide grating device includes at least one waveguide that has a proximal end and a distal end. The distal end of the at least one waveguide has an endface. The device also has a waveguide grating fabricated on the endface of the at least one waveguide. The waveguide grating has at least one waveguide layer and at least one grating layer. The waveguide grating also has a plurality of variable parameters. The plurality of variable parameters includes at least one permittivity of the at least one grating layer, the permittivity of the at least one waveguide layer, the periodic structure of the at least one grating layer, the grating fill factor of the at least one grating layer, the thickness of the at least one waveguide layer, and the thickness of the at least one grating layer. The at least one waveguide layer and the at least one grating layer may be the same layer. Also, the permittivity of the at least one waveguide layer and one of the

permittivities of the at least one permittivity of the at least one grating layer may be the same.

In other respects, the system may also include a source coupled to the proximal end of the at least one waveguide for propagating a signal through the at least one waveguide. After the signal is propagated, it contacts the waveguide grating and is reflected from the waveguide grating in whole or in part, or transmitted through the waveguide grating in whole in or in part, depending at least partially upon the plurality of variable parameters. The source may be a broadband source. The source may be a white light. The source may be a light emitting diode. The source may be a laser. The source may be a continuous wave source. The source may be a pulsed source. The source may be polarized. The source may be unpolarized. The source may be an incoherent light source. The source may be a coherent light source. The source may have wavelengths ranging from the ultraviolet to microwave range (on the order of 100 nm to the order of tens of centimeters).

In still other respects, the system may also include a photodetector operationally coupled to the at least one waveguide. As used herein, if a first device is "operationally coupled" to a second device, one or more mediums or devices may separate the first and second devices such that the first and second devices are not in physical contact with each other. The photodetector may include silicon. The photodetector may include germanium. The photodetector may include indium gallium arsenide. Silicon, germanium, and indium gallium arsenide are examples of semiconductor detectors that may serve as photodetectors operationally coupled to waveguides of the present devices. Semiconductor detectors are power detectors commonly used in the detection of continuous wave sources ranging from about 160 nm to about 1800 nm wavelengths (e.g., visible range to infrared). The photodetector may include a pyroelectric material. The photodetector may include the human eye.

In other respects, the at least one waveguide may be a fiber. The at least one waveguide may be rectangular in shape. The at least one waveguide may be a channel waveguide. The at least one waveguide may be cylindrical in shape. The at least one

waveguide may be a slab waveguide. The at least one waveguide may be a ridge waveguide. The at least one grating layer may include a dielectric material. The at least one grating layer may include a glass. The at least one grating layer may include a polymer. The at least one grating layer may include a liquid or solid crystalline material. The at least one grating layer may include a semiconductor material. The at least one grating layer may include a photorefractive material. The at least one waveguide layer may include a dielectric material. The at least one waveguide layer may include a glass. The at least one waveguide layer may include a polymer. The at least one waveguide layer may include a liquid or solid crystalline material. The at least one waveguide layer may include a semiconductor material. The at least one waveguide layer may include a photorefractive material. The at least one grating layer and the at least one waveguide layer may be the same layer. The at least one grating layer and the at least one waveguide layer may be different layers in contact with each other. The waveguide grating may also include a third layer in contact with the at least one waveguide layer. The third layer may be a buffer layer, which may be formed from any material suitable for forming either the at least one waveguide layer or the at least one grating layer, and which may be formed using the same techniques that may be used to form either the at least one waveguide layer or the at least one grating layer. The third layer may be distinct from both the at least one waveguide and grating layers. The plurality of variable parameters may include the thickness of the third layer. The waveguide grating may also include a third layer in contact with the at least one grating layer, and may include an arbitrarily large number of layers, each of which may be either additional waveguide layers, additional grating layers, or additional buffer layers.

In still other respects, the system may include a sensor operationally coupled to the waveguide grating device. The sensor may be an electrochemical sensor. The sensor may be an optical sensor. The sensor may be a surface plasmon sensor. The sensor may be a fluorescence sensor. The sensor may be an evanescent wave sensor.

In another respect, the invention is a waveguide grating device that includes at least one waveguide through which a signal having at least one wavelength may be propagated. The at least one waveguide has an end, and the end has an endface. The

device also includes a waveguide grating fabricated on the endface of the at least one waveguide. The waveguide grating has at least one waveguide layer and at least one grating layer. The waveguide grating also has a plurality of variable parameters. The plurality of variable parameters includes at least one permittivity of the at least one grating layer, the permittivity of the at least one waveguide layer, the periodic structure of the at least one grating layer, the grating fill factor of the at least one grating layer, the thickness of the at least one waveguide layer, and the thickness of the at least one grating layer. The periodic structure of the at least one grating layer has a period less than the at least one wavelength of the signal. The at least one waveguide layer and the at least one grating layer may be the same layer. Also, the permittivity of the at least one waveguide layer and one of the permittivities of the at least one permittivity of the at least one grating layer may be the same.

In another respect, the invention is a waveguide grating device that includes at least a first waveguide having a first end. The first end has a first endface. The waveguide grating device also includes a first waveguide grating fabricated on the first endface. The first waveguide grating has at least a first waveguide layer and at least a first grating layer. The at least first waveguide layer and the at least first grating layer may be the same layer. The waveguide grating device also includes at least a second waveguide having a second end. The second end has a second endface. The waveguide grating device also includes a second waveguide grating fabricated on the second endface. The second waveguide grating has at least a second waveguide layer and at least a second grating layer. The at least second waveguide layer and the at least second grating layer may be the same layer.

In other respects, the at least first and second waveguides may be fibers.

In another respect, the invention is a method of forming a waveguide grating device that includes providing at least one waveguide that has an end, and the end has an endface; and fabricating a waveguide grating on the endface of the at least one waveguide to form the waveguide grating device.

In other respects, the method may also include cleaving the end to form the endface of the at least one waveguide. The method may also include polishing the end to form the endface of the at least one waveguide.

In still other respects, the waveguide grating may include at least one layer of polymer. The fabricating may include dipping the endface of the at least one waveguide into the polymer. The method may also include heating the at least one layer of polymer. The method may also include patterning the at least one layer of polymer. The patterning may include holographic interferometry, photolithography, electron-beam lithography, laser-beam lithography, or contact printing the at least one layer of polymer to form a grating. The fabricating may include spin coating the endface of the at least one waveguide with a polymer.

In still other respects, the waveguide grating may include at least one layer of photosensitive glass or at least one layer of dielectric. The method may also include etching the at least one layer of dielectric to form a grating.

In other respects, the waveguide grating may include at least a first layer and at least a second layer adjacent the at least first layer. The fabricating may include depositing the at least first layer on the endface of the at least one waveguide by sputtering and coating the at least first layer with the at least second layer. The fabricating may also include depositing the at least first layer on the endface of the at least one waveguide by thermal evaporation. The fabricating may include depositing the at least first layer on the endface of the at least one waveguide by electron-beam evaporation. The fabricating may also include depositing the at least first layer on the endface of the at least one waveguide by molecular beam epitaxy. The fabricating may also include depositing the at least first layer on the endface of the at least one waveguide by metal-organic chemical vapor deposition. The fabricating may include depositing the at least first layer on the endface of the at least one waveguide by chemical vapor deposition. The fabricating may include depositing the at least first layer on the endface of the at least one waveguide by liquid phase epitaxy.

In another respect, the invention is a method of detecting at least one parameter of a medium. As used herein, "medium" means material under investigation in solid, liquid, plasma, or gas form. The method includes providing a waveguide grating device. The device includes at least one waveguide that has an end, and the end has an endface. The device also includes a waveguide grating fabricated on the endface of the at least one waveguide. The waveguide grating has at least one waveguide layer and at least one grating layer. The at least one waveguide layer and the at least one grating layer may be the same layer. The method also includes contacting the waveguide grating with a medium, propagating a signal having at least one signal attribute through the at least one waveguide such that the signal contacts the waveguide grating and the at least one signal attribute is modified, and comparing the modified signal attribute to a known signal attribute to detect the at least one parameter of the medium. As used herein, "signal attribute" means power of a reflected or transmitted wave at a specific wavelength, a specific spectral range, or a specific polarization.

In other respects, the at least one signal attribute may be the spectral content of the signal. The at least one signal attribute may be the intensity of the signal. The at least one signal attribute may be the polarization of the signal. The at least one parameter of the medium may be the presence or absence of a substance. The at least one parameter of the medium may also be the quantity of a substance. The at least one parameter of the medium may be the refractive index of the medium. The at least one parameter of the medium may be the thickness of the medium. The medium may include a first parameter and a second parameter, and the comparing may include comparing the modified signal attribute to a known signal attribute to detect both the first and second parameters.

BRIEF DESCRIPTION OF THE DRAWINGS

The following drawings form part of the present specification and are included to further demonstrate certain aspects of the present invention. The invention may be better understood by reference to one or more of these drawings in combination with the detailed description of specific embodiments presented herein.

FIGS. 1 and **2**. An embodiment of one of the present devices useful as a reflection filter designed for an ionic self assembled polymer waveguide layer having a thickness d_2 and a photoresist grating layer having a thickness d_1 recorded on the top surface. TE polarization at normal incidence, $n_C = 1.0$, $n_{1H} = 1.632$, $n_{1L} = 1.0$, $n_2 = 1.8$, $n_S = 1.45$, $d_1 = 200$ nm, $d_2 = 280$ nm, $\Lambda = 515$ nm, fill factor $f = 0.5$. As depicted, $f\Lambda$ is the width of the high-index region of the grating layer.

FIG. 3. Transmission measurement at normal incidence performed with a broadband source using an embodiment of one of the present devices that has separate waveguide and grating layers.

FIGS. 4 and **5**. Calculated TE and TM-polarization spectral response (**FIG. 4**) of an embodiment of one of the present devices that is useful as a filter and has separate waveguide and grating layers (**FIG. 5**) with the following parameters: $\Lambda = 0.51$ μm, $d_1 = 0.4$ μm, $d_2 = 0.18$ μm, $n_H = 1.63$, $n_L = 1.0$, $n_2 = 1.9$, and $n_S = 1.45$.

FIGS. 6 and **7**. Calculated (**FIG. 6**) and measured (**FIG. 7**) spectral shift of one embodiment of the present waveguide gratings on a planar substrate, before and after immersion in water. Physical parameters are as follows: grating period $\Lambda = 510$ nm, fill factor $f = 0.5$, $n_2 = 2.0$, $d_2 = 200$ nm, $d_1 = 300$ nm, $n_H = 1.62$, $n_L = 1.0$ (air) and $n_L = 1.33$ (water), TE polarization.

FIG. 8. Test setup used to obtain transmission measurements for the present devices used as sensors.

FIG. 9. Schematic of a test setup to measure properties of light (spectrum, polarization, and/or power) from the present waveguide gratings.

FIG. 10. Single beam holographic setup using ultraviolet laser to record grating pattern on optical fiber endfaces coated with photosensitive polymer.

FIG. 11. Raw transmission data measured for an embodiment of one of the present devices having separate waveguide and grating layers that are fabricated using Si3N4 and photoresist, with the following parameters: grating period $\Lambda = 510$ nm,

thickness of the photoresist grating layer, d_1=300 nm, thickness of the waveguide layer (Si3N4), d_2=200 nm, low refractive index of the grating layer, which is the same as the refractive index of the cover region (n_c of air), n_L=1.0, high refractive index of the grating layer, n_H=1.62, refractive index of the waveguide layer (Si3N4), n_2=1.85, refractive index of the substrate (silica optical fiber), n_F=1.45.

FIGS. 12 and **13**. Thickness sensing in water. TE polarization spectral response of an embodiment of the present devices useful as fiber endface reflection filter (**FIG. 13**). The peak wavelength shifts from 749.6 nm to 751.5 nm and 754.1 nm, as 20 nm and 40 nm of material are added, respectively. The physical parameters of the waveguide grating are as follows (**FIG. 12**): grating period, Λ = 454 nm, thickness, d = 371 nm, refractive indices of the grating layer, n = 2.55 (ZnSe) and n = 1.33 (water). The refractive index of the material to be detected is n = 1.4.

FIGS. 14 and **15**. Refractive index sensing in liquid. TE polarization spectral response of an embodiment of the present devices useful as fiber endface reflection filter (**FIG. 15**). The peak wavelength shifts from 749.6 nm to 752.2 nm and 754.8 nm, as the refractive index of the detected liquid varies from 1.33 to 1.34 and 1.35, respectively. The physical parameters of the waveguide grating are as follows (**FIG. 14**): grating period, Λ = 454 nm, thickness, d = 371 nm, refractive indices of the grating layer, n = 2.55 (ZnSe) and n = 1.33 – 1.35 (liquid being detected).

FIGS. 16 and **17**. Thickness sensing in air. TE polarization spectral response of an embodiment of the present devices useful as fiber endface reflection filter (**FIG. 17**). The peak wavelength shifts from 1.554 μm to 1.564 μm and 1.575 μm, as 20 nm and 40 nm of material are added, respectively. The physical parameters of the waveguide grating are as follows (**FIG. 16**): grating period, Λ = 0.907 μm, thickness, d = 1.1 μm, refractive indices of the grating layer, n = 3.2 (Silicon) and n = 1.0 (air). The refractive index of the material to be detected is n = 1.4.

FIGS. 18 and **19**. Thickness sensing in air. TE polarization spectral response of an embodiment of the present devices useful as fiber endface reflection filter (**FIG. 19**). Approximately 1 nm shift for 10 nm of adhered material (n = 1.4). The physical

parameters of the waveguide grating are as follows (**FIG. 18**): grating period $\Lambda = 0.349$ μm, $f = 0.5$, $d_1 = 0.12$ μm, $d_2 = 0.15$ μm, $n_{H,1} = 1.45$ (SiO_2), $n_2 = 2.0$ (HfO_2), $n_{L,1} = n_C = 1.0$, $n_S = 1.45$.

FIGS. 20 and **21**. Refractive index sensing in water. Approximately 3.1 nm shift for 0.01 change in refractive index (**FIG. 21**). The peak wavelength shifts from 807.4 nm to 810.1 nm and 813.3 nm, as the refractive index of the detected liquid varies from 1.34 to 1.35 and 1.36, respectively. Linewidth = 0.8 nm. The physical parameters of the waveguide grating are as follows (**FIG. 20**): grating period $\Lambda = 0.530$ μm, $f = 0.5$, $d = 0.470$ μm, $n_H = 2.0$ (Si_3N_4), $n_S = 1.45$, $n_L = n_C = 1.34$, 1.35, and 1.36.

FIG. 22. Plot of peak wavelength shift for large dynamic range sensing. Response is linear and sensitivity is retained for a refractive index range from 1.3 to 1.7. Corresponds to structure described in **FIG. 20**.

FIG. 23. Scanning electron micrograph of 800 nm period photoresist grating recorded on a multimode fiber endface 800 times magnification.

FIG. 24 (see Appendix). Flow chart of a genetic algorithm using rigorous coupled-wave analysis for merit function evaluation [77]. The program uses the library PGAPACK [110] to perform specific genetic algorithm operations such as mutation, crossover, selection, ranking, and generation of new chromosomes.

FIG. 25 (see Appendix). Crossover and mutation operations illustrated for chromosomes composed of 6 genes encoded as real numbers. In the 3 types of crossover operations shown here genes of the parent chromosomes (white and grey) are exchanged to yield new chromosomes. In the mutation operation, one or more genes are randomly changed from one value to another.

FIGS. 26a and **26b** (see Appendix). Example of a diffractive structure consisting of two gratings in two separate layers, with physical parameters shown in **(a)** and corresponding chromosome represented in **(b)**. The chromosome is a candidate solution in the optimization process. A set of chromosomes forms a population. The total

population of chromosomes at a given iteration is called a generation. In this case, the parameters to be optimized are the grating period Λ, the thicknesses d_1, d_2, refractive indices, $n_{L,1}$, $n_{H,1}$, $n_{L,2}$, $n_{H,2}$, and relative positions of the high-refractive index materials within a grating period, $x_{L,1}$, $x_{H,1}$, $x_{L,2}$, and $x_{H,2}$.

DESCRIPTION OF ILLUSTRATIVE EMBODIMENTS

Disclosed herein is a new GMR device that includes a waveguide having an end that has an endface and a waveguide grating fabricated on the endface. As defined above, waveguides include fibers such as optical, single-mode, multi-mode, polarization-maintaining, graded-index, step-index, nonlinear core (either with or without embedded electrodes), polymer, phototonic crystal waveguides and fibers, glass, crystal-core, and chalcogenide fibers; waveguides with shapes such as rectangular, elliptical and cylindrical; slab waveguides and ridge waveguides. The waveguide grating is made up of at least one waveguide layer and at least one grating layer, and the grating and waveguide layers may be the same layer. The layers, if separate, may be arranged in any suitable fashion with respect to each other and the waveguide. A source may propagate an incident signal, such as a broad-spectrum signal, through the waveguide, the waveguide may guide the signal to the waveguide grating, and the waveguide grating, depending on its design, may filter the signal to reflect or transmit a desired spectral band of the signal. Used as a filter, characteristics such as center wavelength (the wavelength at which a peak or a notch is exhibited in the spectrum of the reflected or transmitted wave), linewidth (the width of the spectral peak or notch) and sideband (reflectance or transmittance in the spectral region outside the peak or notch spectral region) are defined by certain waveguide-grating parameters, such as the periodic structure of the grating layer(s), the refractive indices of the layer(s) forming the waveguide grating, the thicknesses of those layers, and the fill factor of the grating layer(s). The present waveguide grating devices provide a new class of diffractive optical elements as a result of the GMR spectral filters resulting therefrom. Potential applications for the present devices include use as spectral filters for use in fiber optic systems (such as communications), as sensors for high resolution chemical or biochemical sensing, and as integrated polarized reflectors for fiber lasers.

The phrase guided-mode resonance (GMR) refers to a rapid variation in the diffraction efficiency spectrum of waveguide gratings generally, and those described herein. A resonance occurs when an incident wave from a propagated signal that may include more than one wave is phase matched to a leaky guided mode allowed by a waveguide grating. Phase matching may be accomplished through a diffraction grating, which is inherently polarization sensitive. Resonances occurring in subwavelength waveguide gratings (*i.e.*, waveguide gratings having a grating layer(s) with a period, Λ, less than the wavelength, λ, of the input wave admitting only zero-order propagating diffraction orders, where $\Lambda<\lambda/n_s$, λ/n_c where λ/n_s and λ/n_c are the wavelengths in the substrate and cover regions respectively, [*i.e.* regions of propagation of the incident and emerging waves]; λ is the wavelength in vacuum, and n_s and n_c are the refractive indices of the substrate and cover regions, respectively) allow complete energy exchange between the forward and backward propagating zero-order waves. In this case, all higher order diffracted waves are evanescent. In fact, when these evanescent waves correspond to waveguide modes supportable by the WGG, the resonance occurs.

Considering a single layer WGG, for a resonance to occur, the average refractive index of the grating layer, n_{av}, is required to be higher than the refractive index of the surrounding cover and it is required to be higher than the refractive index of the substrate. For a multi-layer structure, one of the layers in the stack needs to meet this requirement. The average refractive index of the grating layer may be calculated using the following equation:

$$n_{av}=[n_L^2 + f(n_H^2-n_L^2)]^{1/2}$$

where n_H and n_L are the refractive indices of the high and low-refractive index regions of the grating layer, and f is the fill factor of the grating layer (*i.e.* the fraction of the grating period occupied by the high-refractive index material). The efficient energy exchange occurs within small ranges of at least one physical parameter of the device, such as the angle of incidence of the input wave or signal, wavelength, thickness of the layers utilized, period of the grating layer(s), and the refractive indices of the grating and waveguide layers and surrounding adjacent media n_f and n_c.

Integration of resonant WGGs with thin-film coatings may provide low sidebands surrounding the resonance regime, achieving high-quality near ideal filter properties. Such filters are disclosed in U.S. Patent No. 5,598,300 to Magnusson and S. S. Wang (1997) (hereinafter the '300 patent), which is hereby expressly incorporated herein by reference in its entirety. Generic GMR filters and their many applications are described in U.S. Patent No. 5,216,680 issued to R. Magnusson and S. S. Wang .

Modeling of the Present Waveguide Grating Devices

Rigorous coupled wave analysis (RCWA) [Gaylord and Moharam, 1985; Moharam, et al., 1995a; and Moharam, et al., 1995b], all three of which are expressly incorporated herein by reference, is a numerical tool that may be used to accurately model the present waveguide grating devices based on the use of certain known parameters of the waveguide grating. "Modeling," as used herein, means to determine the spectral characteristics, *i.e.*, the fraction of the incident wave power that is reflected and transmitted through a waveguide grating device at any wavelength of interest. This includes determining the GMR spectral locations, shape, and width of GMR peaks or notches, and reflectance and transmittance in the sidebands (*i.e.* outside the resonance region). For a rigorous analysis and development of these theories see Magnusson and Wang U.S. Patent No. 5,216,680, which is hereby expressly incorporated herein by reference in its entirety, and [Wang and Magnusson, 1995], which is also hereby expressly incorporated herein by reference in its entirety. However, a simplified model employing waveguide theory is useful to provide physical insight and approximate resonance locations. This theory is detailed in the '300 patent at col. 5, line 6 through col. 6, line 61 for multi-layer waveguide gratings. For a single-layer waveguide grating, this theory is detailed in the '300 patent at col. 7, lines 7 through 38. The present waveguide gratings may be modeled in a variety of different manners. For example, GMR devices may be modeled using a commercially available software such as Gsolver, from Grating Solver Development Company, which implements RCWA. In addition, waveguide gratings may be modeled using finite difference time domain analysis programs such as TFDS, commercially available from Apollo Photonics, or BEAMPROP

FULLWAVE, commercially available from Rsoft. Waveguide gratings may also be suitably modeled using finite element analysis.

The present waveguide gratings formed from multiple layers may be modeled using RCWA. In using such an analysis, one may assume that the grating layer being analyzed possesses an infinite number of grating periods. In using such an analysis, one may also assume the incident wave or waves are plane waves, and further, one may assume the incidence of the plane waves is normal. **FIG. 1** shows a two-layer TE polarization GMR response resulting from an RCWA in which the foregoing assumptions were made. **FIG. 2** depicts a two-layer waveguide grating **10**, which was analyzed to arrive at the response shown in **FIG. 1**. Waveguide grating **10** may be part of a two-layer reflection filter (having waveguide layer **12** and grating layer **14**) designed for an ionic, self-assembled polymer waveguide layer with a photoresist grating layer deposited on the polymer waveguide layer.

Although the assumption that the number of grating periods is infinite may be made in using RCWA to analyze the present waveguide gratings, the number of such grating periods is finite. However, in recent experiments at microwave wavelengths, RCWA has been demonstrated to accurately predict the GMR spectral locations and lineshapes of finite-size structures [Tibuleac, *et al.*, 2000]. Microwave experiments on GMR filters indicate that finite-size grating layers with as few as twelve periods may yield GMR notch filters with a decrease in the transmittance spectrum from ~81% outside resonance to ~2% at resonance. The foregoing GMR filters have, of course, been planar and not fabricated on the end of a waveguide.

Controlling lineshape parameters such as center wavelength, linewidth, and sideband response may be achieved by carefully selecting the parameters of a waveguide grating device. Low sideband responses (*e.g.*, **FIG. 1**) may be achieved by choosing the grating layer thickness to be one-fourth of the resonant wavelength. Filter linewidth is affected in part by the strength of the guided-mode confinement and the coupling efficiency of the waveguide grating. As used herein, "mode confinement" means the

ratio between the power contained in the core of a mode and the power contained in the core and cladding of a mode. Lineshape may be adjusted by modifying the grating layer fill factor, the grating modulation, and the difference between the average refractive index of the grating layer and the refractive indices of the surrounding regions or media. As used herein, "grating modulation" means the difference between the high and low refractive indices of the grating layer. The filter central wavelength is affected in part by the periodic structure of the grating layer (as used herein, "periodic structure of the grating layer" includes grating layer parameters such as grating period, shape of the gratings, the dimension of the gratings, amplitude, and periodicity of the gratings), the refractive indices of the grating layer, and the refractive index of the waveguide layer (or refractive indices if more than one waveguide layer is utilized within the waveguide grating). Additional parameters that may be manipulated to affect lineshape include the relative spatial phases of incorporated gratings, which may be shifted, and the periodic structures of any grating layers, which may be chosen to be dual-line [Tibuleac, 1996]. As used herein, "dual-line" refers to the presence of two peaks (or notches) in the spectral reflectance (or transmittance) dependence. Since waveguide grating devices are polarization dependent in 1-D (*i.e.*, one-dimensional) grating layers, polarization insensitive devices may be designed by implementing crossed (*i.e.*, 2-D) waveguide gratings.

The present waveguide grating devices may also be designed an inverse approach if certain properties of the needed GMR device are known. If the properties of the needed GMR device are known, these parameters may serve as input parameters into a search and optimization algorithm such as a genetic algorithm [Goldberg, 1989] employing RCWA to calculate the reflectance and transmittance spectra of the devices during the optimization process. Such methods to design GMR filters have been reported in references [Tibuleac, *et al.*, 1997; Zuffada, *et al.*, 1998; Zuffada, *et al.*, 1999; Tibuleac, 1999]. The use of one such genetic algorithm is disclosed in the Appendix hereto.

Biomedical/chemical sensing

A biosensor is an analytical device that integrates an immobilized biologically sensitive material, such as enzyme, antibody, DNA, cells, or organic molecules, with an electrochemical, piezoelectric, optical or acoustic transducer to convert a biochemical response into a signal that can be used for measurement, interpretation, or control. Accurate, real-time, direct measurement of biologically related substances eliminates expensive and complex sample preparation that is required in *ex situ* lab processing. Electrochemical and optical sensors are the most widely used and versatile biosensing methods [Collings and Caruso, 1997; Kersey, 1996]. Optical biosensors provide a fast, accurate, safe, and robust means of analyte detection. All fundamental characteristics of light as it interacts with matter can be used in measurement, including intensity, frequency, phase, and polarization changes. A major advantage of optical detection methods over other techniques is the ability to probe surfaces and films in a non-destructive manner. In addition, optically based sensors are generic elements that can be used to sense a wide variety of analytes that might not be possible with other methods, such as gases, proteins, various types of micro-organisms, and metabolites such as glucose. The use of optical fibers in biosensing systems allows a high degree of geometrical versatility, including component miniaturization, and continuous, real-time, remote monitoring of very small sample domains. Optical fiber sensors are convenient devices that are free from electrical interference and are generally biocompatible for *in vivo* testing. The ability to provide remote, continuous monitoring is a distinct advantage when testing hazardous materials, *in vivo* testing, or down-well environmental measurements.

Current fiber optic sensor technology includes fluorescence [Golden, *et al.*, 1994; Abel, *et al.*, 1996], total internal reflection fiber sensors [Bolin, *et al.*, 1989], reflection intensity [Tugendhaft, *et al.*, 1997; Jin, *et al.*, 1997; Johns, *et al.*, 1998], surface plasmon resonances [Jorgenson and Yee, 1993; Jung, 1997; Furlong, *et al.*, 1996a; Slavik, *et al.*, 1997b; Slavik, *et al.*, 1997a; Slavik, *et al.*, 1998; Homola and Slavik, 1996; De Maria, *et al.*, 1993; Melendez, *et al.*, 1997], and fiber bundle arrays utilizing fluorescent detection

materials [Ferguson and Walt, 1997]. While fiber optic sensors are the focus here, there are many other designs in the area of optical sensing. Capillary optical sensors utilize fiber optic couplers and capillary tubes that are chemically modified on the inner surface. Optical absorbance or fluorescence is implemented as the unit of measure. Ellipsometry is used to detect refractive index or thickness changes in biological sensing layers. Sensors utilizing planar optical waveguides [Collings and Caruso, 1997; Melendez, *et al.*, 1996; Sharma and Rogers, 1994] include total internal reflection fluorescence, attenuated total reflectance, reflectomeric interference spectroscopy, as well as thin film devices including the resonant mirror developed by Cush, *et al.* [Cush, *et al.*, 1993], grating couplers [Sychugov, *et al.*, 1997], and Mach-Zender sensor devices [Luff, *et al.*, 1998]. Fabrication of optical sensor elements using transparent sol-gel, can increase sensor sensitivity [Cunningham, 1998]. The primary advantage of fiber optic sensing over other optical configurations is the real-time, remote operation of the sensor.

Current technology

The surface plasmon resonance (SPR) is a widely used optical detection method that is highly sensitive to changes in the optical properties at the sensor surface, such as refractive index or thickness. The term surface plasmon (SP) is based upon an electromagnetic field charge-density oscillation that can occur at the surface of a conductor. When this electromagnetic coupled mode of excitation travels along the interface between a metal and another medium, it is referred to as a surface plasmon. These surface waves are bound to the metal-dielectric interface, with an intensity maximum in the surface and exponentially decaying fields perpendicular to it. An SP mode is resonantly excited by TM polarized incident light if the wavevector of the incident light and the surface plasmon wave are matched, as governed by Maxwell's equations. At resonance, reflected light intensity from the metallic surface goes through a minimum at a defined angle of incidence. Phase matching occurs by employing a metallized diffraction grating, or by using total internal reflection from a high index material, such as in prism coupling or an evanescent field from a guided wave. The propagation constant of the plasmon depends upon the refractive index of the adjacent medium, which is within sensing distance of the surface plasmon field.

Conventional surface plasmon sensors include a prism or diffraction grating that is used as the phase matching and transducer element. Commercial [Sethi, 1994] planar SPR sensors include Pharmacia Biosensor's BIAcore and BIAlite systems, and Texas Instrument's Spreeta system [Melendez, *et al.*, 1997; Furlong, *et al.*, 1996b; Ouellette, 1998]. Fiber optic SPR sensors developed by Jorgenson and Yee [Jorgenson and Yee, 1993], and more recently by Slavik, *et al.* [Slavik, *et al.*, 1997b; Slavik, *et al.*, 1997a; Slavik, *et al.*, 1998] and Jung [Jung, 1997] allow remote, real time monitoring. Commercially manufactured fiber SPR sensors are available from Biacore. A sensor based upon SP wave excitation on the tip of an optical fiber was proposed by De Maria, *et al.* in 1993 [De Maria, *et al.*, 1993].

A method for fabrication of fiber optic surface plasmon resonance sensors is described by Jorgenson and Yee, and Slavik, *et al.* This includes removal of the fiber cladding over the sensing region to allow access to the evanescent field of a guided mode. Cladding removal is accomplished by gluing the fiber in a curved slot on a silica block, and subsequently polishing and lapping the cladding to obtain a proximity to the core. The exposed region is covered with a thin layer of gold in order to support an SP wave, with the sensing layer attached at the outer interface. If the two modes are closely phase matched, a guided TM mode in the fiber can excite an SP wave at the outer metal-sensing layer interface, resulting in a detectable minimum in the transmitted light intensity. The wavelength where this intensity minimum occurs is closely dependent on the refractive index of the medium adjacent to the metallic film (sensing layer). Variations in the sensing layer, such as refractive index or thickness, can be detected by monitoring changes in the output intensity. To attain maximum sensitivity, appropriate metal layer and cladding thicknesses must be chosen. Optimizing the sensor sensitivity by increasing metallic layer thickness and decreasing the remaining fiber cladding thickness results in a decrease of the dynamic range of the sensor. To tune the refractive index operation range of the SPR fiber optic sensor, a thin tantalum pentoxide overlayer can be deposited beneath the sensing layer.

Slavik details two modes of operation for the fiber optic SPR sensor [Slavik, *et al.*, 1997a]. In the spectral mode, the output power is monitored as a function of

wavelength to indicate an SPR spectral location. A tunable laser or a white light source can be used in this configuration. Experimentally achieved sensor resolutions operating in the spectral mode are reported to be $1.6*10^{-5}$ RIU (refractive index units) for an index range of 1.3952-1.3973. This is based on a spectroscopic resolution of 0.1 nm, and does not account for the lineshape characteristics of the sensor response. In the more widely reported amplitude mode, the output power is monitored at a fixed wavelength. The relative output intensity is detected as a refractive index or thickness change causes a shift towards or away from a resonance location. Small changes in the transmitted intensity are measured and calibrated to a specific refractive index or thickness change. Resolutions for the amplitude mode are reported as low as 9×10^{-6} RIU for an index range of 1.4105-1.4163, assuming an optoelectronic system that can resolve changes in optical power to 1%. A particular intensity response provides two solutions for a change in refractive index or thickness; one going towards the resonance dip and one away from it. The resolutions reported are based on an experimentally determined resonance shift of approximately 12 nm with a 80 nm linewidth for a refractive index change from 1.3952 to 1.3973. To perform remote, real-time measurements with this sensor, an aluminum mirror must be deposited on the fiber endface to redirect the output light.

Other fiber sensors include fluorescent excitation and detection in the evanescent field of an optical fiber. As the evanescent field extends to the cladding sensing region, fluorophores coated on the outside of the fiber are excited. Depending on the biological material that is being sensed, a particular wavelength of light indicating a biological recognition can be captured by the fiber optic probe and analyzed using spectroscopic detection elements. This requires very sensitive detection equipment such as photomultiplier tubes since the captured signal is very weak. Accuracy and repeatability is an inherent weakness for this device. Furthermore, detection applications are limited by the bioselective agents available.

Advantages of the present waveguide grating devices over current technology

Comparison of biosensor performance is best accomplished through a figure of merit [Cunningham, 1998]. Device characterizations that can be included in a generic figure of merit include sensor sensitivity, resolution, and dynamic range. The sensitivity of a biosensor is defined as the measured response for a particular amount of material that is detected. For example, a GMR resonance shift of 11 nm for a thickness change of 20 nm results in a sensitivity value of 0.55 nm shift per 1 nm material added. However, the sensitivity value does not consider sensor limitations; rather, it indicates the maximum achievable sensitivity to the analyte being detected. Resolution of the sensor includes the realistic component limitations such as spectroscopic equipment resolutions, power meter sensitivities, bioselective agent response, and linewidth considerations. The lineshape response has a great impact on the accuracy of spectroscopic sensors in distinguishing between wavelength shifts. For highest confidence limits, the resolution for resonant sensors can be defined by the linewidth (full width, half maximum power), assuming the equipment has a higher resolution. For example, the refractive index sensor depicted in **FIG. 20** has a maximum resolution of $3*10^{-4}$ RIU, when considering only spectroscopic resolution limitations of 0.1 nm. However, the detected response is limited by the linewidth of the device, which is 0.8 nm in this case. Under this criteria, the smallest RIU change that can be accurately detected with this sensor is $4*10^{-3}$, although the sensitivity is higher. By normalizing the resonance shift with the linewidth, a realistic evaluation of a resonance sensor performance can be determined. A comparison of sensor sensitivity, resolution and dynamic range is included below in Table 1. The dynamic range (or usable range) of a sensor is defined as the range where discrimination between responses can be detected. Materials used in fabrication and the sensing medium generally limit this range. In addition, these three parameters are generally inter-related. For example, for SPR sensors, as the operational range of refractive index values that can be detected is increased, the sensor sensitivity decreases.

Sensor type	Max. Linewidth Response	Max. Sensitivity	Max. Resolution (equipment resolution 0.1 nm)	FOM: Sensor Resolution (w/ linewidth)	Dynamic range
SPR sensor	80 nm [Slavik, et al., 1997a] 30 nm [Slavik, et al., 1998]	6250 nm/RIU 1875 nm/RIU	$1.6 * 10^{-5}$ RIU $5.3 * 10^{-5}$ RIU	$1.3 * 10^{-2}$ RIU $1.6 * 10^{-2}$ RIU	1.352-1.3973 not available
Fiber GMR sensor (**FIG. 20**)	0.8 nm 1.7 nm	310 nm/RIU 330 nm/RIU	$3 * 10^{-4}$ RIU $3 * 10^{-4}$ RIU	$2.6 * 10^{-3}$ RIU $5.1 * 10^{-3}$ RIU	1.34-1.36 RIU 1.3 – 1.7 RIU

Table 1. Comparison between the surface plasmon sensor and guided-mode resonance fiber sensor.

The present devices (e.g., the fiber GMR sensors in Table 1) can be highly sensitive to the parameters of the waveguide gratings. Thus, the grating period, filling factor, number of layers, layer thicknesses, and refractive indices may be tailored for a specific waveguide grating device sensitivity and operational dynamic range. This flexibility allows the resonance wavelength, linewidth, and degree of sensitivity to be tailored for specific applications. By using a genetic algorithm program to design the present devices, specific design criteria such as sensing range or sensitivity may be realized. In general, the present waveguide grating devices have a much higher operational sensing range and greater sensor sensitivity than other fiber optic sensors. In addition, by utilizing biologically sensitive material, such as biopolymers to fabricate the waveguide grating, increased waveguide grating sensitivity may be achieved.

Low loss dielectric materials may be used as either the grating layer or the waveguide layer of the present devices, and absorption losses are not a physical limitation of the present devices. Linewidths for the present devices may be typically less than 5 nm, with well-defined resonance shapes that may provide accurate, well-defined measurements. Two separate resonance locations for TE and TM polarizations are

available for detection in the present devices. Accordingly, accuracy and reliability of the present devices is greatly enhanced over other sensors types, since each polarization can act as a reference for the other. In addition, it may be possible to obtain actual refractive index and thickness values of the sensed medium since two measured values are obtained. In contrast to that which may be achieved using the present devices, for sensors that utilize only TM polarization, such as surface plasmon resonance (SPR) sensors, the refractive index of the sensed medium or the sensed layer thickness must be determined beforehand, since both parameters cannot be determined from the same measurement.

Current fiber optic sensors, including the SPR, require the sensing region to be along the length of the fiber, which increases fabrication complexity and spatial sensing resolution. For the present devices, however, the sensing element is located on the waveguide endface, such as the endface of a fiber, thus permitting highly-accurate, small-proximity sensing. Furthermore, waveguide sensor arrays, such as optical fiber sensor arrays, may be readily implemented to simultaneously detect a wide variety of analytes, such as DNA sequences. A calibration fiber may be integrated in a bundle of the present devices to further increase accuracy for *in vivo* or remote measurement.

Current fiber optic array sensors utilize fluorescence indicators and are less sensitive than the present devices. Additionally, since the deposition of dielectric thin films on optical fiber endfaces is well-known in the art, the present devices are suitable for mass production. Moreover, an array of the present devices may be fabricated simultaneously using standard thin film deposition methods well known in the art such as dipping, sputtering, spin coating, thermal evaporation, electron-beam evaporation, molecular beam epitaxy, metal-organic chemical vapor deposition, chemical vapor deposition, and liquid phase epitaxy, and submicron grating fabrication technology such as contact printing, and patterning techniques well known in the art such as holographic interferometry, photolithography, electron-beam lithography, and laser-beam lithography. Further still, other detection devices and techniques, such as SPR sensors or fluorescent detection, may be combined with the present devices, which utilize the GMR effect, to increase the flexibility of the present devices in a system for spectral filtering.

Applications for the present devices and systems include use as fiber optic sensors for chemical/biochemical measurement in widespread applications that range from implantable devices used for continuous *in vivo* measurement to *ex vivo* analysis in a laboratory. Additionally, in fiber optic communications, the present device may be used to reject or transmit signals for multiplexing/demultiplexing of multiple wavelength channel systems. It is also to be understood that the present waveguide grating devices include multiple waveguides having ends with endfaces, on each of which waveguide gratings may be fabricated. Thus, the present devices may be used as sensors having multiple waveguides with waveguide gratings fabricated on the respective ends thereof. The present device also includes a waveguide, such as an optical fiber, having an end with an endface on which a waveguide grating is fabricated, which waveguide is adjacent to a second waveguide, such as an optical fiber, having an end with an endface onto which a waveguide grating may be fabricated. The waveguide gratings on the two waveguides may be oriented such that a signal propagated through the first waveguide may be reflected at least in part after contacting the first waveguide grating such that it then contacts the waveguide grating of the second waveguide and, thereafter, may be reflected by the second waveguide grating such that the signal is then transmitted through the second waveguide in a direction moving away from the second waveguide grating. In such an embodiment, the present device is a dual fiber sensor. Some applications include:

Feedback control in artificial organs;

Dynamic intravascular blood gas sensor used to detect oxygen saturation of hemochromes (hemoglobin, myoglobin) and carbon dioxide levels in major blood vessels or cardiac chambers. Specifically, for the detection of cardiac shunts during catheterization, to estimate cardiac output from arterio-venous oxygen difference, or for use in the care of fetuses to determine oxygen saturation data. Inadequate blood oxygen levels and carbon dioxide elimination are indications of respiratory and metabolic imbalances. By continuous, real-time monitoring of these levels in the blood, dynamic corrections to patient oxygen ventilation or pharmacological agents can be administered;

Glucose sensor used to detect blood/tissue glucose levels. Qualitative measurement based on refractive index differences correlated to glucose concentration levels can be used. A more accurate quantitative measurement is made by employing bioselective agents such as glucose oxidase. Glucose oxidase changes its chemical properties (and refractive index) depending on the concentration of glucose available in the detection sample;

pH sensor used to monitor blood/tissue acidity levels can be implemented by employing a pH sensitive biosensitive layer on the fiber endface GMR device that changes refractive index for different blood acidity levels;

Tumor sensors to assist in surgery for tumor removal based on an increase in refractive index from the cancer cells; and

Brain tissue sensors to locate neuro structures for guidance during neurosurgery. As the refractive indices of tissues vary in the gray and white matter of the brain, the present device may act as a sensor to distinguish between the two types of tissues. For similar reasons, the present devices may also be used as sensors for detecting brain tumors or lesions, *etc.*

Fuel tank sensor to detect the level of a liquid in a container, or the density or composition of gases inside a fuel container based on changes in the refractive index of the medium.

Oil/fuel quality sensor to detect changes in the chemical properties that induce refractive index changes in the oil or fuel. For example, one of the present devices could be used as a real-time sensor in an automobile that detects when engine oil needs replacement.

Setup for measuring spectral reflectance from present devices

As explained herein and in more detail in the Examples below (e.g., Example 3), the present devices are suitable candidates for use as spectroscopic sensing elements (i.e., filters) due at least in part to the narrow linewidths and high sensitivities of the guided-

mode resonance peaks to variations in the physical parameters of the present devices (layer thicknesses, refractive indices, grating fill factor, and substrate and cover refractive indices). Thus, the present devices may be designed for a specific sensitivity, resolution, and operational dynamic range. This flexibility allows the resonance wavelength, linewidth, and degree of sensitivity to be tailored for specific applications. A sensor may be optimized to enhance sensitivity to specific parameters, such as refractive index and/or thickness. Since dielectric materials may be used in the fabrication of the present devices, many design configurations are available. In **FIG. 9,** one embodiment of a setup for use in monitoring the reflectance from one of the present devices is depicted. In general, the setup may be used to measure properties of light (spectrum, polarization, and/or power) reflected from the present devices The setup includes an input light source **30**, a 2x2 optical coupler **20,** and a detection unit that is not illustrated. Input light source **30** enters 2x2 coupler **20** at input port **22,** and coupler **20** divides the power of light incident from input port **22** between output ports **26** and **28**. As depicted in **FIG. 9,** output port **26** is coupled to one of the present devices, which includes a waveguide **16** (such as a fiber) on which waveguide grating **10** is disposed. The reflected power **34** from the present device is equally split between input port **22** and port **24**. Port **24** is coupled to a detection unit, which measures the properties of light reflected from the present device. To monitor the intensity of the reflected signal **36** using a fixed wavelength laser source, the detection unit may be an optical power meter, such as a Newport 835 optical power meter. Alternatively, to monitor spectral shifts of the GMR resonance, the detection unit may be an optical spectrum analyzer (such as an Anritsu MS9001B), or a monochromator (Burleigh model) and an optical power meter. Input light source **30** may be broadband (such as an LED or white light source), or a tunable laser (such as Ti:Sapphire or a semiconductor type). To detect the polarization state of the reflected signal **36**, an optical polarizer should be placed so that it receives reflected signal **36** prior to the detection unit. The setup depicted in **FIG. 9** permits various medium parameters to be monitored remotely and in real-time.

The present device includes multiple sensors, at least one of which is a waveguide having an end with an endface on which a waveguide grating is fabricated, bundled in an array, such that many types of sensors may be simultaneously utilized. A potential

application is to integrate this type of bundled array with an intravenous (IV) tube that is inserted into a patient's artery. By integrating the biosensor array into the polymer shunt used for insertion of the IV tube, real time, accurate, continuous monitoring of blood gases, glucose and pH levels can be accomplished without loss of patient blood. This is particularly useful for monitoring changes in blood gas/glucose levels during surgery, or critically ill patients in intensive care units.

The following examples are included to demonstrate specific embodiments of the invention. Example 1, however, does not include a description of an embodiment of the present devices. Instead, it includes a description of fabricating a diffractive grating (i.e., a grating layer) on a fiber endface. As the diffractive grating is not also a waveguide layer, it is not a waveguide grating. However, the description accompanying Example 1 may be useful in creating the present devices because the waveguide gratings of the present devices require a grating layer. Further, were the photoresist in Example 1 fabricated on a fiber having a slightly lower refracting index, the photoresist could have served as both a waveguide layer and a grating layer, thereby forming a waveguide grating. It should be appreciated by those of skill in the art that the techniques disclosed in the examples that follow represent techniques discovered by the inventors to function well in the practice of the invention, and thus can be considered to constitute exemplary modes for its practice. However, those of skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention. See also [Wawro, 1999] for information identical or similar to the following Examples.

EXAMPLE 1

Example 1 describes a procedure that was undertaken to optimize the manner of fabricating a diffractive grating, but not a waveguide grating, on the endface of a fiber. A high quality cleaving tool was used to obtain flat, optical quality endfaces on both single-mode and multimode fibers. After cleaving, the fibers were visually inspected with an optical microscope to determine endface quality. The fibers were cleaned by immersion

in an ultrasonic bath of heated acetone for 30 minutes, briefly dipped in optical grade methanol, and dried with filtered nitrogen gas.

The fabrication procedure was initially optimized by recording efficient submicron diffractive structures on the optical fiber endfaces. Thin films of UV-sensitive Shipley 1805 photoresist (PR) were deposited on the cleaved multimode and single-mode fiber endfaces by a dipping process. It was found that a dilution of 3 parts PR to 5 parts Shipley photoresist thinner yielded an approximate PR thickness of 300 nm. This thickness value was obtained from a test substrate that was dipped in the thinned solution, after which the thickness of the PR layer was measured via ellipsometery. The PR/thinner mixture was filtered before deposition with a glass fiber 0.2 μm particulate filter. No significant curvature of the PR layered on the fiber endfaces was observed in scanning electron microscope pictures for photoresist dilutions of greater than 1:1. After the dipping process, the fibers were soft baked in an oven at 90°C for approximately 30 minutes. The soft bake improved PR adhesion and response linearity during exposure.

Diffraction gratings with submicron periods were recorded in the PR layers using holographic interferometry with an UV Argon ion laser (λ = 365 nm) as illustrated in **FIG. 10**. An exposure power of approximately 110 $\mu W/cm^2$ for 22 seconds was required. The gratings were developed for approximately 20 seconds using Shipley MF-321 developer, resulting in a surface relief photoresist grating.

The grating diffraction efficiency was tested by propagating laser light into the uncoated end of the fiber, and measuring the power output of the transmitted diffraction orders on the end with the diffraction grating. Light from each diffracted order was collimated with a lens and measured individually with an optical power detector head to obtain accurate intensity measurements. The intensity output of the transmitted diffracted orders was measured after coupling a white light source (halogen lamp) into an optical fiber with a 1.2 μm period photoresist grating recorded on its endface. An HeNe laser light (λ=633 nm) was also propagated into the optical fiber. A diffraction grating with 800 nm period on an optical fiber endface with a 100 μm core diameter was also evaluated, and is illustrated in **FIG. 23**. This device produced ±1 diffracted orders

containing ~50% of the total output power when tested with a HeNe laser (λ=633 nm). In addition, gratings with a period of 530 nm were recorded on optical fiber endfaces with 6.7 μm core diameters. The ±1 transmitted diffraction orders were measured to contain ~10% of the total power coupled out of the fiber at a wavelength of 442 nm (HeCd laser).

EXAMPLE 2

Fabrication of Waveguide Gratings on Endfaces of Waveguides

In this example, waveguide gratings were fabricated on the endfaces of optical fibers. Once the ends of the fibers were cleaved to form endfaces and cleaned, deposition of dielectric thin-films was required to create a waveguide grating structure. In this example, thin films of Si_3N_4 were deposited by sputtering on the clean, uncoated optical fiber endfaces. Silicon nitride is a hard, low loss dielectric material that has a relatively high refractive index (n=2.0). This commonly-used coating can also be patterned by etching in a reactive ion etching (RIE) chamber using standard fluorocarbon etchant gases, such as CF_4 or CHF_3. An RF-powered sputter machine that housed a single, three-inch Si_3N_4 target was used to deposit the nitride films. Inert argon gas was used as the primary sputter gas, with a small amount (~5%) of N_2 included to prevent nitrogen depletion of the Si_3N_4 target. Nitrogen depletion results in an Si-rich film, which is typically quite lossy. The fibers were mounted in the chamber along with a test substrate made from fused silica to monitor the thickness of the deposited nitride films. The thickness, refractive index, absorption, index grading, and surface roughness of the deposited films were measured using a Woollam V-Vase spectroscopic ellipsometer.

Next, the test substrate was spin coated with a 300 nm thick layer of PR and a 510 nm grating was recorded on its surface. Using the spectroscopic ellipsometer, the test waveguide grating structure was subjected to normal incidence transmission measurements, the results of which are indicated in **FIG. 3**. The parameters of the test waveguide grating structure that led to the results depicted in **FIG. 3** are shown therein. The ellipsometer testing source is a fiber coupled Xenon arc lamp monochrometer with a specified resolution of 0.1 nm.

EXAMPLE 3

FIG. 4 depicts calculated TE and TM polarization spectral responses of a waveguide grating with the cross section shown in **FIG. 5**, having the following parameters: grating period, Λ, is 0.51 μm; thickness, d_1, is 0.4 μm; thickness, d_2, is 0.18 μm; refractive index, n_H, is 1.63; refractive index, n_L, is 1.0; refractive index, n_2, is 1.9; and refractive index, n_S, is 1.45. The calculations leading to the results depicted in **FIG. 4** were performed with rigorous coupled-wave analysis, assuming plane waves at normal incidence on a structure with an infinite number of grating periods.

Turning now to the details of this experiment, ~200 nm layers of Si_3N_4 were sputter deposited on multimode optical fiber endfaces with 100 μm core diameters. PR gratings with 510 nm periods were subsequently recorded to yield waveguide grating devices. The parameters of the devices are as follows: grating period, L, is 510 nm; PR thickness, d_1, is 300 nm; Si_3N_4 thickness, d_2, is 200 nm; refractive index, n_L, is 1.0; refractive index, n_H, is 1.85; Si_3N_4 refractive index, n_2, is 1.85; fiber refractive index, n_s, is 1.45.

Testing was performed using the setup depicted in **FIG. 8**. Spectral measurements made with tunable Ti:Sapphire laser (λ=730–900 nm) indicated GMR notches of ~18% in the transmitted power, which was measured at the output of the optical fiber. **FIG. 11** illustrates the measured results without normalization. The low efficiency is partially attributed to the polarization sensitivity of the GMR effect, with TE and TM peaks occurring at different wavelengths and the polarization scrambling induced by propagation through the optical fiber. However, similar devices that are polarization independent may be achieved with two-dimensional gratings. Scattering due to imperfect fiber cleaves and rough silicon nitride films are also contributing factors to a decrease in GMR efficiency. Furthermore, it is assumed for modeling purposes that the wavefronts are essentially planar in nature due to the large core diameter. More accurate modeling may be required to account for the finite 2-D confinement of the incident beam, as well as the finite periodic structure on the fiber endface. It is contemplated that finite element or finite difference modeling would be well-suited for this purpose.

Spectroscopic Sensor Designs

The present waveguide grating devices are well-suited for use as spectroscopic filters due to the sensitivity of the devices to changes in parameters such as the thicknesses of the grating layer or layers and the waveguide layer or layers, the refractive indices of the same, the grating fill factor, and the substrate and cover refractive indices. Factors in addition to the parameters discussed above that may affect the configuration of a given waveguide grating device include the sources available for testing and the required sensor resolution. In general, waveguide gratings made of a single layer (*i.e.*, waveguide gratings in which the at least one waveguide layer and the at least one grating layer are the same layer) are more sensitive to changes in the parameters discusses above than are waveguide gratings made of multiple layers, because the mode confinement of the single-layer waveguide grating is greater or heightened as compared to the mode confinement of multi-layer waveguide gratings.

FIGS. 12-22 illustrate examples of the present waveguide grating devices that may be used as filters/sensors for sensing changes in the parameters of the refractive index and the thickness of material that may contact the waveguide grating. The devices may be placed and utilized in fluid media including water and air.

EXAMPLE 4

Sensor Placed in Aqueous Media and Used to Sense Changes in Thickness of Material Deposited on Waveguide Grating

FIG. 12 illustrates certain parameters of both a sensor designed to detect changes in the thickness parameter of a material in an aqueous media, and a material contacted by the sensor. The waveguide grating is made of ZnSe and is fabricated on an endface of the waveguide, which, in this embodiment, is an optical fiber. Grating period, Λ, is 454 nm, thickness, d, of the waveguide grating is 371 nm, refractive index, n_{wg}, of the waveguide grating is 2.55, and refractive index, n_{water}, of water is 1.33. The refractive index of the material to be detected is 1.4. Material is a high index material can be deposited on waveguide grating by plasma etching. The above refractive index and thickness values were chosen to model typical bioselective agents, such as antigen/antibody attachments.

FIG. 13 illustrates the TE polarization spectral response of the waveguide grating device illustrated in **FIG. 12** to material and to changes in the thickness of material. As shown, a resonance shift of 1.9 nm was determined as 20 nm of material was added to the thickness of waveguide grating, and a 2.6 nm resonance shift was determined as a total of 40 nm of material was added to the thickness of waveguide grating as the peak wavelength shifted from 749.6 nm, to 751.5 nm, to 754.1 nm, respectively. In this case, the degree of resonant central wavelength shift is contributed to two parameter changes: the change in waveguide grating thickness that resulted from adding material to the waveguide grating, and a change in grating layer (which is also the waveguide grating) fill factor. The former parameter may contribute significantly to the resonance shift for fill factor values other than 0.5 [Tibuleac, 1996].

EXAMPLE 5

Sensor Placed in Liquid Media and Used to Sense Changes in Refractive Index of Liquid

Turning now to **FIGS. 14** and **15**, the above highly flexible sensor configuration may also be used to detect changes in the refractive index of a media into which it may be placed. The media was liquid, and the refractive index of the liquid changed from 1.33 to 1.35. Accordingly, **FIG. 15** illustrates the TE polarization spectral response of the waveguide grating device illustrated in **FIG. 14** to the changes in the refractive index of the liquid. As shown in **FIG. 15**, the peak wavelength shifted from 749.6 nm to 752.2 nm and 754.8 nm as the refractive index of the detected liquid varied from 1.33 to 1.34 and 1.35, respectively.

EXAMPLE 6

Sensor Placed in Liquid Media, Air, and Used to Sense Changes in Thickness of Material Deposited on Waveguide Grating

In **FIGS. 16** and **17**, a waveguide grating device is shown that may be placed in a media of air. The device may be contacted by a material, which in this case, was deposited on waveguide grating. The device shown was used as a sensor to detect

changes in the thickness of material. **FIG. 16** illustrates the parameters of the device and material. Waveguide grating is made of Si and is fabricated on endface of waveguide which, in this embodiment, is an optical fiber. Grating period, Λ, is 0.907 μm, thickness, d, of waveguide grating is 1.1 μm, refractive index, n_{wg}, of waveguide grating is 3.2, and refractive index, n_{air}, of air is 1.0. The refractive index, $n_{material}$, of the material to be detected is 1.4. **FIG. 17** illustrates the TE polarization spectral response of the waveguide grating device illustrated in **FIG. 16** to material and to changes in the thickness of material. As shown in **FIG. 17**, the peak wavelength shifted from 1.554 μm to 1.564 μm and 1.575 μm, as 20 nm and 40 nm of material were added, respectively. Due to the higher index modulation in this sensor design, a resonance shift of 10 nm per 20 nm change in thickness is available for sensing. The incident wavelength was in the range of 1.55 μm, which corresponds to tunable laser diode wavelengths.

EXAMPLE 7

Sensor Placed in Liquid Media, Air, and Used to Sense Changes in Thickness of Material Deposited on Double-Layer Waveguide Grating

A waveguide grating device with a double-layer waveguide grating is depicted in **FIG. 18**. The depicted design is useful for thickness sensing in air using a visible incident light source. **FIG. 18** illustrates certain parameters of both the device and the material deposited on the waveguide grating of the device. The waveguide grating is composed of a grating layer made of SiO_2 in contact with a waveguide layer made of a HfO_2, which is fabricated on endface of waveguide. Grating period, Λ, is 0.349 μm, fill factor, f, of grating layer is 0.5, thickness, d_1, of grating layer is 0.12 μm, thickness, d_2, of waveguide layer is 0.15 μm, refractive index, $n_{H,1}$, of grating layer is 1.45, refractive index, n_2, of waveguide layer is 2.0, refractive index, $n_{L,1}$, is 1.0, as is refractive index, n_C, and refractive index, n_S, is 1.45. **FIG. 19** illustrates the TE polarization spectral response of the waveguide grating device illustrated in **FIG. 18** to material and to changes in the thickness of material. As shown, a resonance shift of approximately 1 nm was determined for each 20 nm of material added to the thickness of grating layer.

EXAMPLE 8

Sensor Placed in Liquid Media and Used to Sense Changes in Refractive Index of Liquid

A waveguide grating device with a highly sensitive single-layer waveguide grating is depicted in **FIG. 20**. The depicted design is useful for refractive index sensing in liquid. This sensor was fabricated using Si_3N_4 as the waveguide grating and may be used to detect small or large changes in the refractive index of a liquid. **FIG. 20** illustrates certain parameters of the device. Waveguide grating is made of Si_3N_4 which is fabricated on endface of waveguide. Grating period, Λ, is 0.530 μm, fill factor, f, of waveguide grating is 0.5, thickness, d, of waveguide grating– is 0.470 μm, refractive index, n_H, of the waveguide grating is 2.0, and refractive index, n_S, of the substrate is 1.45. Refractive index, n_L, of the liquid being sensed is the same as refractive index, n_C, of cover region, both of which are determined to range from 1.34 to 1.36. **FIG. 21** illustrates the TE polarization spectral response of the waveguide grating device illustrated in **FIG. 20** to liquid and to changes in the refractive index of material. As shown, a resonance shift of approximately 3.1 nm was determined for a change in refractive index of liquid of 0.01. The peak wavelength shifted from 807.4 nm to 810.1 nm and 813.3 nm, as the refractive index of the detected liquid varied from 1.34 to 1.35 and 1.36, respectively. Also shown in **FIG. 21**, the linewidth is 0.8 nm.

FIG. 22 illustrates the extremely large dynamic range and linear response available for the sensor design depicted in **FIG. 20**. **FIG. 22** is a plot of the reflectance peak wavelength shift that occurred as the refractive index of the detected liquid increased over a large range of refractive index values. The response depicted in **FIG. 22** is relatively linear and sensitivity may be maintained for a refractive index range from 1.3 to 1.7. As illustrated by these figures, high sensitivity is maintained for both refractive index ranges, making this design a very attractive and flexible sensor design.

All of the compositions and/or methods and/or apparatus disclosed and claimed herein may be made and executed without undue experimentation in light of the present disclosure. For example, techniques associated with preparing the endfaces of the present

waveguides such as cleaving and polishing are known in the art. Techniques associated with fabricating, including dipping and spin coating, heating, and etching, and techniques associated with depositing, including sputtering, thermal evaporation, electron-beam evaporation, molecular beam epitaxy, metal-organic chemical vapor deposition, chemical vapor deposition and liquid phase epitaxy, are known in the art. Techniques associated with patterning, including holographic interferometry, photolithography, electron-beam lithography, laser-beam lithography, and contact printing, are known in the art.

While the compositions and methods of this invention have been described in terms of specific embodiments, it will be apparent to those of skill in the art that variations may be applied to the compositions and/or methods and/or apparatus and in the steps or in the sequence of steps of the method described herein without departing from the concept, spirit and scope of the invention. For example, it is contemplated that waveguide gratings may be fabricated on graded index lenses. Additionally, the waveguide gratings of the present devices may be fabricated on the endfaces of the waveguides by directly recording the grating pattern into a dielectric material such as glass, for example. In such an embodiment, a photosensitive polymer for patterning or etching the grating pattern into the dielectric would not be used. Such fabrication may be realized through, for example, stresses that may be induced by illumination of the dielectric with a laser or an electron beam. For example, chalcogenide glass forms a surface relief grating if exposed to a laser interference pattern. Additionally, the present waveguide gratings may be fabricated on electro-optic waveguides. For example, an electro-optic fiber (such as one commercially available from Sentel Technologies) fabricated from a nonlinear dye-doped polymer having electrodes embedded around the fiber core may be prepared as described above to have an endface on which a waveguide grating may be fabricated. Since the refractive index of the core of such a fiber changes upon the application of a voltage, the resonance transmission or reflection peak may change depending on the refractive index of the core. In such an embodiment, the effect just described may be useful in calibrating the device or tuning it to a specific wavelength band (tunable filter). More specifically, it will be apparent that certain agents that are both chemically and physiologically related may be substituted for the agents described herein, while the same or similar results would be achieved. For example, dielectric

materials from which the grating and waveguide layers of the present devices may be made include nonlinear dielectric materials. For example, polymers that incorporate nonlinear materials (for second order or third order nonlinear effects) or semiconductor materials having nonlinear materials therein may be utilized for the present layers. Consequently, present devices having waveguide gratings with such nonlinear materials may be used as wavelength converters (second harmonic generation). All such similar substitutes and modifications apparent to those skilled in the art are deemed to be within the spirit, scope and concept of the invention as defined by the appended claims.

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